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Modeling the Environmental Effects on Carbon Fibers in a Ceramic Matrix at Oxidizing Conditions

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July 2000

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MODELING THE ENVIRONMENTAL EFFECTS ON CARBON FIBERS IN A CERAMIC MATRIX AT OXIDIZING CONDITIONS

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ABSTRACT

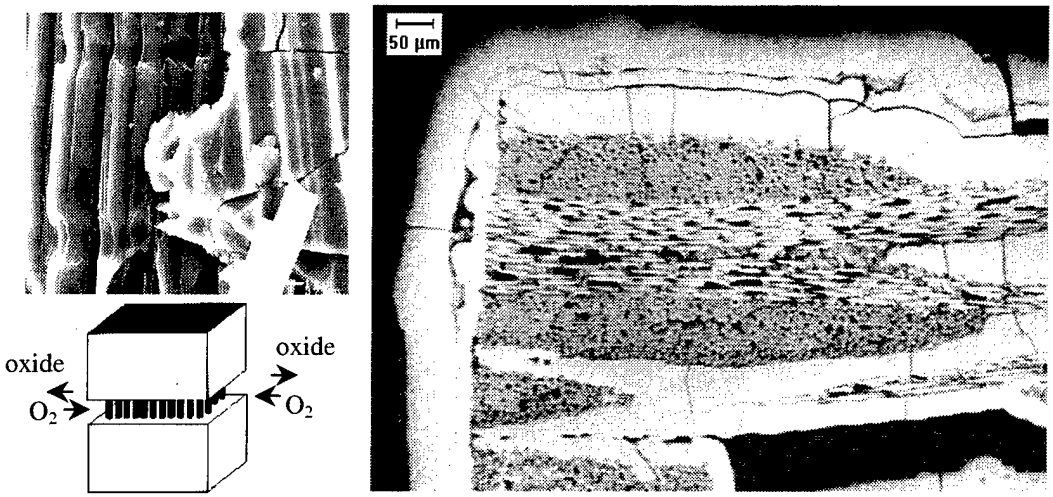
Carbon fiber/silicon carbide matrix composites (C/SiC) are promising materials for space applications such as nozzles, thrusters, and bladed disks. However the as-fabricated, cracked nature of the material makes it susceptible to environmental degradation due to fiber oxidation. Theoretical models can be used to gain a better understanding of how certain variables (i.e. temperature, environment, diffusion coefficient and reaction rate constant) are related to a specific process and to supplement or analyze experimental results. Methods for predicting the distribution of local oxygen concentration from the edge to the interior of an open column in a matrix bounded by carbon fiber tows will be discussed. The amounts and distribution of oxygen in a matrix and the gradients in oxygen concentration convey information regarding the oxidation kinetics. The models consider the case of a crack in a ceramic matrix that is bridged by carbon fiber tows. Two analytical solutions and differing finite difference models will be used to predict oxygen concentrations. Results from each prediction method will be presented and compared. The results and their relation towards oxidation kinetics will be discussed. Several geometries for the composite (square and rectangular) are considered in the finite difference models.

INTRODUCTION

Ceramic matrix composites (CMCs) are a relatively new class of materials. Since these materials are still in their infancy compared to such materials as metals, large databases on the properties of CMCs have not been developed. Other factors such as material properties changing as the material is continuously developed and the costs associated with processing and testing CMCs have hindered the development of large databases. Modeling the properties of materials can aid in the understanding of material properties, reduce material development costs, and extend data bases by simulating experimental results and then predicting material properties at conditions beyond those considered in experimental testing.

Under the Advanced Space Transportation Program task STR-56, Ceramic Matrix Composite (CMC) Life Prediction, extensive material testing and models are being adapted as design tools to aid in the application of CMC material systems into flight experiments. The material that will be focused on is carbon fiber reinforced silicon carbide matrix composites (C/SiC). The program is being monitored by the NASA Marshall Space Flight Center. The life prediction tasks are being conducted at the NASA Glenn Research Center. This paper will focus on one task within this program: modeling the environmental effects on the reinforcing carbon fibers within a non-reactive matrix. Greater detail on the overall scope of the life prediction program can be found elsewhere [1].

The as-fabricated microcracks in C/SiC materials provide an avenue for oxygen ingress that leads to the oxidation of the carbon fibers within the cracked matrix. The cracks form upon cooling down from the processing temperature due to stresses that arise from the difference in the thermal expansions of the carbon fibers and the silicon carbide matrix. The crack bridging carbon fibers within C/SiC materials that have been exposed to high temperature oxidizing conditions are observed to oxidize along the portions of the fibers that bridge the microcracks as illustrated in Figure 1a. In polished cross-sections of C/SiC material, microcracks can be traced from edge to edge within the polished section as the crack traverses through the matrix and fiber tows (Figure 1b). Based on these and similar observations from microstructural analysis, the model will assume the simplified case, shown in Figure 1c, of a completely cracked matrix that is bridged by an array of fiber tows.



Figures 1a-1c. 1a-Fracture surface from a stressed oxidation (creep rupture) test showing how the matrix cracks provide diffusion paths for oxygen that reacts with portions of the fiber that bridge the crack (upper left). 1b-Cracks traversing the interior of a composite material consisting of a 0/90 fiber tow weave (right). 1c-Illustration of simplified crack bridging scenario assumed in the models (lower left).

THEORY

The oxidation rate or the rate at which carbon recedes will depend on both the reaction rate constant and the diffusion coefficient. The reaction rate constant (K [m/s]) correlates to the reactivity of the carbon while the diffusion coefficient (D [m^2/s]) correlates to the supply of oxygen. Both of these processes, carbon/oxygen reactions and oxygen supply, occur in series and have a temperature dependence. The more dominating of these two steps has the potential to control or limit the overall process of carbon recession. A dimensionless parameter, called the Sherwood number which often appears in oxidation kinetics equations, is used to correlate the reaction rate constant with the diffusion coefficient such that

$$Sh = \frac{K\,\Delta x}{D} \tag{1}$$

The term Δx [m] is a characteristic length taken as one-half the column width in our case (Δx=a).

The reaction rate constant depends on such factors as the activation energy, impurity level, surface porosity, and crystal structure of the carbon as well as temperature [2,3]. The diffusion coefficient depends on such factors as total pressure, molecular weight, force constant and collision diameter for the product and reactant gases, and temperature [4]. When all factors relating to the material and environment are held constant and only temperature is changed in the range of 600°C to 1500°C, the effect of temperature on the reaction rate constant and diffusion coefficient in an oxygen environment can be realized. The reaction rate constant has a strong temperature dependence and can change by approximately three orders of magnitude while the diffusion coefficient only changes by three times across this temperature range [5]. Depending on the value of these two variables, the Sherwood number will have either a relatively high or relatively low value. The value of the Sherwood number will correlate to either diffusion controlled kinetics, reaction controlled kinetics, or mixed control kinetics.

Illustrations at two stages of the oxidation process within a 2-D cross-section for each of the two extreme types of kinetics and representative microstructures are shown in Figure 2. For reaction controlled kinetics, which occurs in the lower temperature range, there will be a general oxidation that occurs throughout the interior of a given cross-section. This oxidation pattern is due to the carbon reactions occurring at a slower rate than the rate at which the oxygen diffusing in through cracks can be consumed. The interior of the composite will be saturated in a high concentration of oxygen that is there to supply the relatively slow carbon/oxygen reactions. In the related micrograph, oxidation occurs primarily at the edge and along microcracks, however some oxidation along microcracks within fiber tows is seen deep into the material. For diffusion controlled kinetics, which occurs at high temperatures, there will be a moving reaction front of carbon. The carbon is very reactive and consumes oxygen as soon as it is supplied so that the interior becomes deprived of oxygen. A shrinking core effect is seen as the reaction front moves inward. There will be a sharp gradient in oxygen concentration that is high at the edge where it first

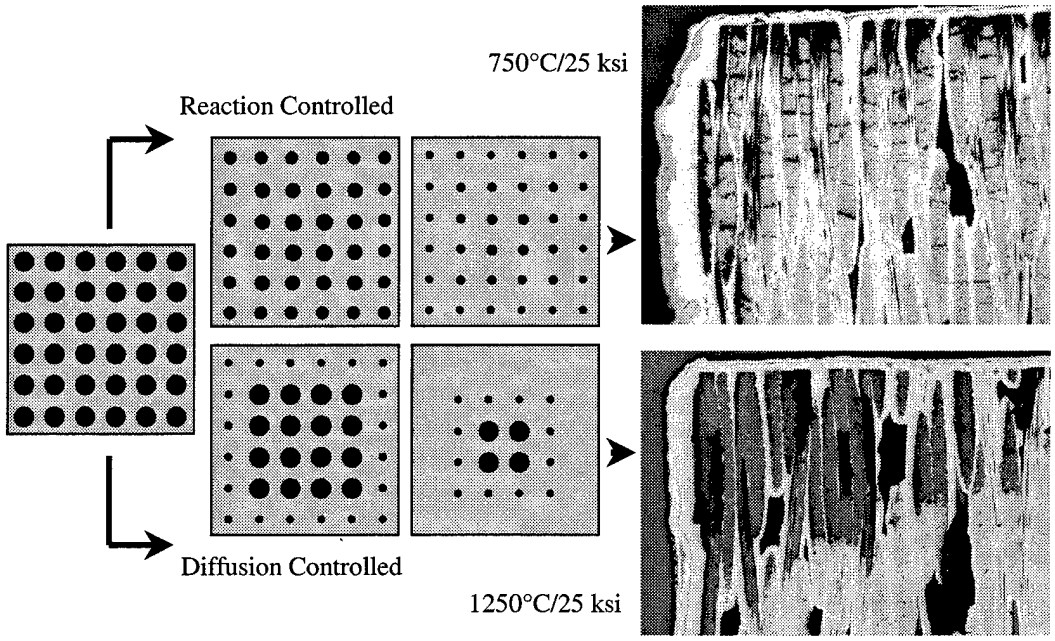


Figure 2. Cross section illustrations of the oxidation patterns at two stages of oxidation in a 6x6 array of carbon fiber tows for reaction controlled (top middle) and diffusion controlled kinetics (bottom middle). Example polished cross-sections of microstructures show the two types of kinetics as obtained from stressed oxidation (creep rupture) tests conducted in air at an elevated temperature under a constant tensile stress until failure (right).

diffuses inward and is very low or zero at the carbon surface. The related micrograph shows the position of the reaction front at the time of failure, which occurred during a stressed oxidation (creep rupture) test. The already consumed side of the reaction front will have a steeply decreasing gradient in oxygen concentration up to the carbon surface while the unconsumed side of the reaction front is deprived in oxygen.

MODELING

Analytical solutions and finite difference methods were used to model the diffusion of oxygen from an external edge to the interior of a cracked composite. The basis for the models is the illustration in Figure 1c. The models consider a cross section that is bridged by fiber tows. Cross-sections of the actual cases considered in the models are shown in Figure 3a-e. Analysis of the oxygen concentrations along the midline (dashed line) for each model will allow the different models to be compared.

In the finite difference models, a similar method to that used by Glime and Cawley [6] is used for determining the local oxygen concentration for each grid. In this method, the oxygen concentration of a grid is determined by sampling the concentration from its four neighboring grids. Certain equations are used depending on the number of neighboring grids that contain carbon and the number of grids that are in the open region where gas phase diffusion occurs. Inputs/variables in the model include the oxygen concentration along the external boundary, and the Sherwood number, which encompasses the variables of temperature, reaction rate constant, and diffusion coefficient. The oxygen concentration at the edge was arbitrarily set at 1 mol/cc so that internal concentrations can be interpreted as a fraction of the concentration at the edge. A calculation of the local oxygen concentration is done for every grid in the mesh pattern. The process is then repeated over hundreds of iterations until a steady-state in oxygen concentration is reached. The result is the local oxygen concentration for each grid in the mesh pattern. Local oxygen concentrations and gradients in overall oxygen concentration across the section can be used to interpret the oxidation kinetics. Greater detail of finite difference model development can be found in previous papers [7,8].

In the finite difference model method for determining oxygen concentrations in the interior of a cross section, grids are used to map out the cross sections. Figures 3a-3c are cross sections of unidirectional fiber tows that make up the bridging arrays. They are used to illustrate the 12 x 12, 12 x 24, and 12 x 36 fiber tow array finite difference models (the 12x12, 12x24, and 12x36 FTA models respectively). Fiber tows represent the bundles of fibers that

provide strength and shape to the composite material. Carbon fiber tows are often in bundles of 1000 individual fibers. An individual fiber has a diameter of approximately 8 microns. In Figure 3a-3c, fiber tows are 10 grids in diameter and are spaced 10 grids from one another and from the edge. It should be noted that Figure 3 only illustrates the boundaries, fiber tows and open bridged regions. Detail of the square mesh patterns are not shown. The finite difference models are used to calculate the steady-state oxygen concentration for the diffusion of oxygen from the external edge to the interior of the sample. The models assume a continuous supply of oxygen from the edge and a continuous supply of carbon from the fiber tow array. In the analysis, only the initial steady-state condition is considered. The steady-state condition will provide information about the kinetics.

Figure 3d illustrates the case of diffusion of oxygen down a single column bounded by fiber tows whose profiles extend into the column. In this model, called the bumped wall finite difference model (BW model) the outer walls are no flux boundaries which allow the model to consider the case of an infinitely wide fiber tow array (i.e., 12 x infinity). There is no diffusion of oxygen into the column from the sides. The resulting oxygen concentration in the column arises only from the diffusion of oxygen from the open ends into the column. Again the fiber tows are 10 grids in diameter and they are spaced 10 grids apart. The column is 250 grids long. Results from the BW model will be compared to the 12x12, 12x24, 12x36 FTA models in order to determine when large geometries (i.e. 12x36) can be simplified to the BW model.

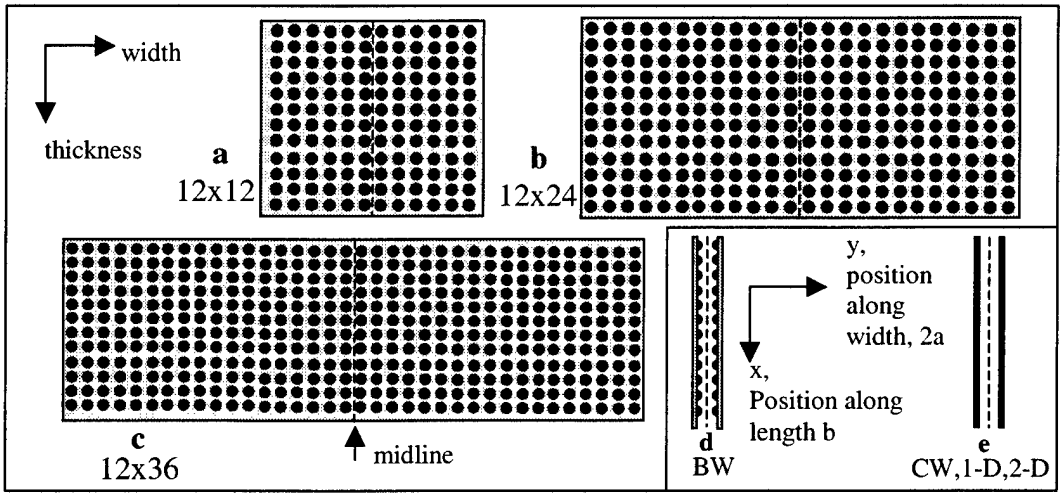


Figure 3a-e. Cases considered in the models. Cross-sections of arrays of fiber tows that bridge a crack in a matrix are shown in Figure 3a-c. Figures a, b, and c illustrate the 12 x 12, 12 x 24 and 12 x 36 FTA models of bridging reactive carbon fiber tows. Figure 3d illustrates the BW model of a single column bounded by fiber tow profiles evenly spaced on each side with no-flux boundary walls. Figure 3e is a single column bounded by reactive carbon walls (used for the CW model and the 1-D and 2-D analytical solutions)

The final scenario to be considered in the analysis is for the case represented in Figure 3e. This is for the diffusion of oxygen down the length of a single column (250 grids) that is bounded by walls of reactive carbon spaced 10 grids apart and is open for diffusion from the ends. A finite difference model, called the continuous wall finite difference model (CW model), was used to determine the oxygen concentration at steady-state. One-dimensional (1-D) and two-dimensional (2-D) analytical solutions were also used to calculate the oxygen concentrations at steady-state. Results from the CW model will be compared to the 1-D and 2-D analytical solutions in order to see how well the finite difference method compares to theoretical mass transport solutions.

The 1-D analytical solution for the oxygen concentration in a column bounded by reactive carbon walls, as illustrated in Figure 3e, is

$$C(x) = \frac{C_1 \sinh\left[(Sh/a)^{1/2}\,(2b - x)\right] + C_2 \sinh\left[(Sh/a)^{1/2}\,x\right]}{\sinh\left[(Sh/a)^{1/2}\,(2b)\right]} \qquad (2)$$

Equation (2) correlates with Carslaw and Jaeger's [9] 1-D solution for heat transfer and is similar to the mass transport equation used by Geankoplis [4]. The oxygen concentration at each end is C_1 and C_2 respectively and they are equal in our case. The position along the length is x and the column length is b. Note that Equation (2) has a term for the total width of the rod, where a is one-half the width. However the equation does not allow for oxygen concentration to be determined at different positions along the width, position y. Therefore this solution is only good for small cross-sections since the oxygen concentration at a given position along 2a is the same for all points along the width.

The 2-D analytical solution for the oxygen concentration in a column bounded by reactive carbon walls, as illustrated in Figure 3e, is

$$C(x,y) = 2\frac{Sh}{a} C_o \sum_{n-1}^{\infty} \frac{\cos(\alpha'_n\, y/a)\ \cosh[(\alpha'_n/a)(b-x)}{\{[(\alpha'_n/a)^2 + ((Sh/a)^2)a] + Sh/a\}\cos(\alpha'_n)\cosh((\alpha'_n\, b/a)} \tag{3}$$

The equation includes roots (α') to the following trancendental equation where

$$\alpha' \tan \alpha' = Sh \tag{4}$$

Equation (4) is a variation in the transcendental equation used by Carslaw and Jeager [9] in their derivation for the 2-D heat transfer equation. Their transcendental equation was multiplied by a, one-half the column thickness or Δx, and $a\alpha$ was substituted by α' such that

$$a\alpha \tan a\alpha = \alpha' \tan \alpha' = fa = Sh \tag{5}$$

where

$$\alpha' = a\alpha \text{ and } f = K/D. \tag{6, 7}$$

The 2-D mass-transport solution represented in Equation (3) is similar to Carslaw and Jaeger's [9] 2-D heat transfer solution. The oxygen concentrations are the same at both ends, C_o. It is seen that this 2-D solution allows for variations in the position, y, along width, 2a. Geometries that have larger cross-sections can be considered. Thus local oxygen concentrations for specific positions and gradients in oxygen concentration can be determined in both the x and y directions.

RESULTS AND DISCUSSION

The steady-state oxygen concentrations for the 12x12, 12x24, 12x36 FTA models, the BW model and the CW model were determined by the finite difference method. The 1-D and 2-D analytical solutions and CW model were calculated for the reactive wall case as illustrated in Figure 3e. The finite difference models and analytical solutions (1-D and 2-D) were compared by calculating oxygen concentrations along the midlines of each model/analytical solution as illustrated by the dashed lines in Figure 3a-e.

Resulting oxygen concentrations along the midline for the case of the reactive wall case, illustrated in Figure 3e, were analyzed. The oxygen concentrations determined by the CW model, the 1-D analytical mass transfer solution, and the 2-D analytical mass transfer solution were compared. All three methods gave a very similar prediction in oxygen concentration for high Sherwood numbers which correlate to the reaction controlled regime and low temperatures. At low temperatures, the carbon reacts slowly so that the column can become saturated in oxygen before it is consumed. However, for higher Sherwood numbers, i.e. Sh>0.1, the 1-D analytical solution predicts a higher gradient in oxygen concentration than for the midlines of the CW model and 2-D solution. At high Sherwood numbers, the carbon is very reactive and the oxygen is therefore more quickly depleted. Because of the high carbon reactivity, there will not only be a significant gradient in oxygen concentration along the length of the column, but also across its width. Since the 1-D solutions are the same for all positions along a given width (midpoint of width to carbon wall), the predicted oxygen contrations along the column are the same throughout the column for all lines from the midline to along the line directly adjacent to and parallel to the carbon wall.

The 2-D analytical solution and the CW model allowed the oxygen concentration along any line for the 5 grid positions of a (one-half the width) to be determined. The oxygen concentrations for the five lines going from the midline to the line parallel to and adjacent to the carbon wall, are shown in Figure 4 for one-eighth of the distance into the column length. The CW model and the 2-D solution compared very well with one another in both specific oxygen concentrations and in trends. At low Sherwood numbers, all oxygen concentration curves fall on one another due to the relatively fast diffusion process and the slow reaction process. However, at high Sherwood numbers, the

effect of position along the width can be seen. There is more of a gradient for the curve directly adjacent to the carbon wall compared to the midline. In each packet of five curves for each Sherwood number, the curve with the lowest gradient in oxygen concentrtion is for the midline while the curve with the largest gradient in oxygen concentration is along the line directly adjacent to the carbon wall. Thus, at high Sherwood numbers, gradients in oxygen concentration are seen along the length and also across the width of the column. The one curve predicted by the 1-D solution for each Sherwood number appears in Figure 4 as the tick marked line.

The oxygen concentrations across the midlines for the FTA models (illustrated in Figure 3a-c) and the BW model (illustrated in Figure 3d) were compared. The oxygen concentrations across the midline for several Sherwood numbers are shown in Figure 5. At low Sherwood numbers the narrower fiber tow array models (12x12 and 12x24 FTA models) gave higher oxygen concentrations due to the reaction process being slow enough for oxygen to diffuse into the considered column (dashed lines in Figure 3a-c) from both directions: along the thickness and along the width. However when the array is sufficiently wide, ie. 12x36, diffusion in from the sides is not a factor in the midline results and the results match closely with the case for an infinitely wide array (12 x infinity or BW model). At high Sherwood numbers, the width of the fiber tow array makes little difference in the resulting oxygen concnetration. The carbon is so reactive that oxygen is quickly depleted and is not able to diffuse very far into the matrix in high concentrations.

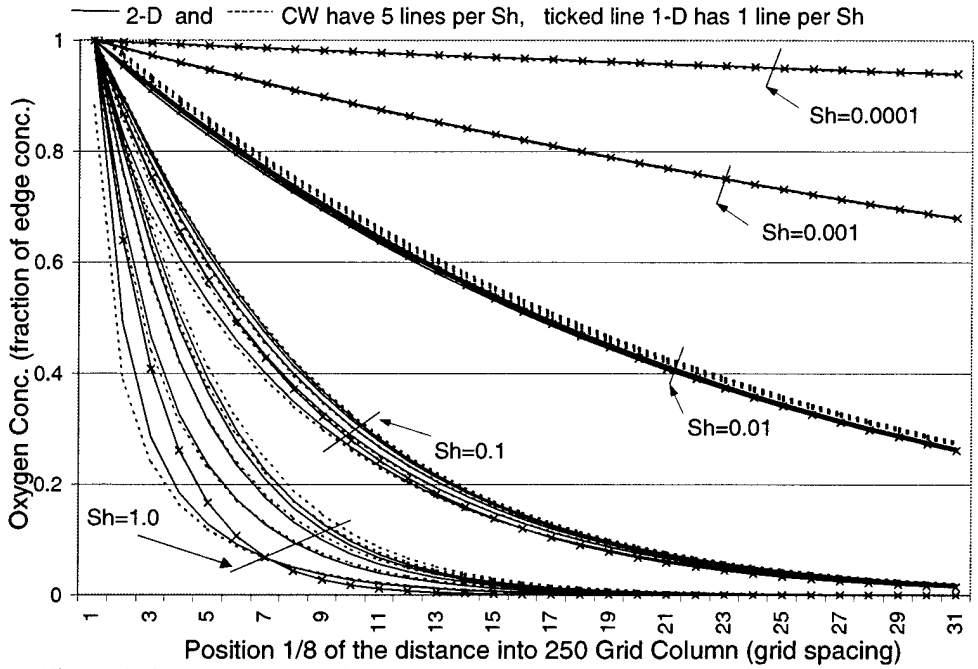


Figure 4. Oxygen concentrations one-eighth of the distance down the column length for the CW model and the 2-D analytical solution for the five lines of width parallel to the midline. Concentrations for each Sherwood number are shown. Gradients in the x and y directions are seen. Also the one line of oxygen concentration predicted by the 1-D model is shown.

CONCLUSIONS

Finite difference models and analytical solutions can be used to calculate the concentration of oxygen in a cracked matrix in which oxygen diffusion and carbon reactions are occurring. Results from the finite difference method (specifically the CW model) agreed well with the analytical solutions for mass transport. The 1-D analytical solution for a column bounded by continuous reactive carbon walls agreed well with the CW model and the 2-D analytical solution at low Sherwood numbers. However it was not accurate at high Sherwood numbers where the high reactivity of carbon also causes gradients in oxygen concentration along the width of the column. For the FTA finite difference models, it was found that when the width is three times greater than the overall thickness (i.e. 12x36), the solution for the midline is much like that for the case of a column with no flux walls. The column with no flux walls represents the case for an infinitely wide fiber tow array (i.e. BW model which correlates to 12 x infinity). The relation between the large fiber tow array model and the much simpler column is very important

in analysis. The large arrays can require thousands of iterative calculations that are quite time consuming especially when analysis includes actual carbon loss over hundreds of small time steps. When studying diffusion and oxidation kinetics in ceramic matrix composites of sufficient width and thickness, the BW model can allow for much simpler and quicker analysis of the oxidation kinetics.

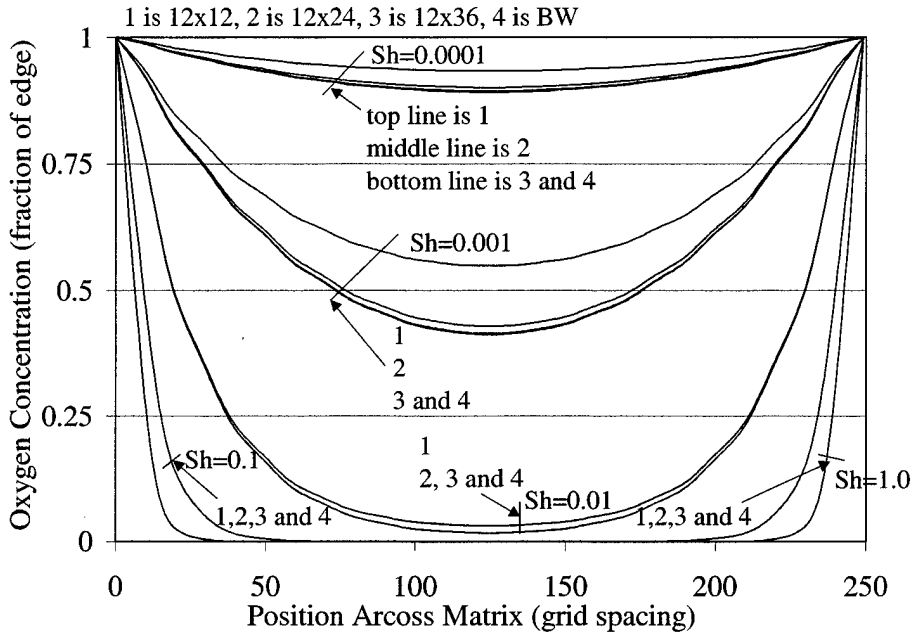


Figure 5. Oxygen concentrations across the dashed midlines shown in Figure 3a-3d for the 12x12, 12x24, and 12x36 FTA models and the BW model.

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13. ABSTRACT ***(Maximum 200 words)***

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